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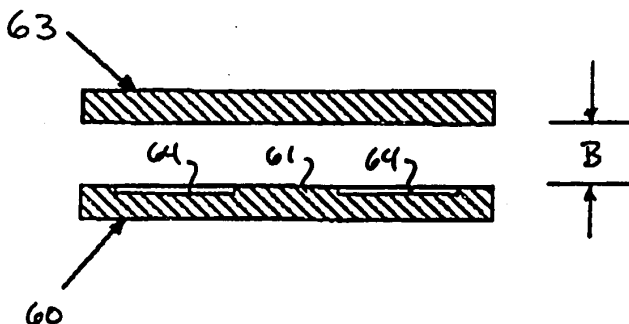
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(54) Title: SUSCEPTOR DESIGNS FOR SILICON CARBIDE THIN FILMS

(57) Abstract

A susceptor is disclosed for minimizing or eliminating thermal gradients that affect a substrate wafer during epitaxial growth. The susceptor includes a first susceptor portion including a surface for receiving a semiconductor substrate wafer thereon, and a second susceptor portion. The susceptor is characterized in that the second susceptor portion faces the substrate receiving surface and is spaced from the substrate-receiving surface, with the spacing being sufficiently large to permit the flow of gases therebetween for epitaxial growth on a substrate on the surface, while

small enough for the second susceptor portion to heat the exposed face of a substrate to substantially the same temperature as the first susceptor portion heats the face of a substrate that is in direct contact with the substrate-receiving surface.



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## SUSCEPTOR DESIGNS FOR SILICON CARBIDE THIN FILMS

### Field of the Invention

The present invention related to semiconductor manufacturing processes,  
5 and in particular relates to an improved susceptor design for epitaxial growth on silicon carbide substrates.

### Background of the Invention

The present invention relates to the production of epitaxial layers of semiconductor materials on silicon carbide substrates. Silicon carbide offers a  
10 number of advantageous physical and electronic characteristics for semiconductor performance and devices. These include a wide bandgap, high thermal conductivity, high saturated electron drift velocity, high electron mobility, superior mechanical strength, and radiation hardness.

As is the case with other semiconductor materials such as silicon, one of  
15 the basic steps in the manufacture of a number of silicon-carbide based devices includes the growth of thin single crystal layers of semiconductor material on silicon carbide substrates. The technique is referred to as "epitaxy," a term that describes crystal growth by chemical reaction used to form, on the surface of another crystal, thin layers of semiconductor materials with defined lattice  
20 structures. In many cases, the lattice structure of the epitaxial layers (or "epilayers") are either identical, similar, or otherwise related to the lattice structure of the substrate. Thus, epitaxial growth of either silicon carbide epitaxial layers on silicon carbide substrates or of other semiconductor materials on silicon carbide substrates, is a fundamental technique for manufacturing devices based on silicon  
25 carbide.

Silicon carbides, however, a difficult material to work with because it can crystallize in over 150 polytypes, some of which are separated from one another by very small thermodynamic differences. Furthermore, because of silicon carbide's high melting point (over 2700°), many processes for working silicon carbide,  
30 including epitaxial film deposition, often need to be carried out at much higher temperature than analogous reactions in other semiconductor materials.

Some basic reviews of semiconductor manufacturing technology can be found for example in Sze, Physics of Semiconductor Devices, 2d Ed. (1981), Section 2.2, pages 64-73; or in Dorf, The Electrical Engineering Handbook, CRC Press, (1993) at Chapter 21 "Semiconductor Manufacturing," pages 475-489; and particularly in Sherman, Chemical Vapor Deposition for Microelectronics: Principles, Technologies and Applications, (1987), ISBN 0-8155-1136-1. The techniques and apparatus discussed herein can be categorized as chemical vapor deposition (CVD) or vapor phase epitaxy (VPE) in which reactant gases are exposed to an energy source (e.g., heat, plasma, light) to stimulate a chemical reaction, the product of which grows on the substrate.

There are several basic techniques for CVD epitaxial growth, the two most common of which are the hot (heated) wall reactor and cold wall reactor processes. A hot wall system is somewhat analogous to a conventional oven in that the substrate, the epitaxial growth precursor materials, and the surrounding container are all raised to the reaction temperature. The technique offers certain advantages and disadvantages.

The second common conventional technique is the use of a "cold wall" reactor. In such systems, the substrate to be used for epitaxial growth is placed on a platform within a container (typically formed of quartz or stainless steel). In many systems, the substrate is disc-shaped and referred to as a "wafer." The substrate platform is made of a material that will absorb, and thermally respond to, electromagnetic radiations.

As is known to those familiar with such devices and techniques, the susceptor's response to electromagnetic radiation is an inductive process in which alternating frequency electromagnetic radiation applied to the susceptor generates an induced (inductive) current in the susceptor. The susceptor converts some of the energy from this inductive current into heat. In many systems, the electromagnetic radiation is selected in the radio frequency (RF) range because materials such as glass and quartz are transparent to such frequencies and are unaffected by them. Thus, the electromagnetic radiation passes through the container and is absorbed by the susceptor which responds by becoming heated,

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along with the wafer, to the temperatures required to carry out the epitaxial growth. Because the container walls are unaffected by the electromagnetic energy, they remain "cold" (at least in comparison to the susceptor and the substrate), thus encouraging the chemical reaction to take place on the substrate.

5       A thorough discussion of the growth of silicon carbide epitaxial layers on silicon carbide substrates is set forth for example in U.S. Patents Nos. 4,912,063 to Davis et al. and 4,912,064 to Kong et al., the contents of both of which are incorporated entirely herein by reference.

10       The use of a cold wall reactor to carry out epitaxial growth, although satisfactory in many respects, raises other problems. In particular, because a semiconductor wafer rests on a susceptor, the wafer side in contact with the susceptor will become warmer than the remainder of the substrate. This causes a thermal gradient in the axial direction through the wafer. In turn, the difference in thermal expansion within the wafer caused by the axial gradient tends to cause the  
15       peripheral edges (typically the circumference because most wafers are disc-shaped) to curl away from, and lose contact with, the susceptor. As the edges lose contact with the susceptor, their temperature becomes lower than the more central portions of the wafer, thus producing a radial temperature gradient in the substrate wafer in addition to the axial one.

20       These temperature gradients, and the resulting physical effects, have corresponding negative affects on the characteristics of the substrate and the epitaxial layers upon it. For example, if the edges are placed in extreme tension, they have been observed to crack and fail catastrophically. Even if catastrophic failure is avoided, the epitaxial layers tend to contain defects. At silicon carbide  
25       CVD growth temperatures (e.g., 1300° - 1800°C), and using larger wafers (*i.e.*, two inches or larger), wafer bending becomes a significant problem. For example, Figure 3 herein plots the values of wafer deflection (H) at various axial temperature gradients as a function of the wafer diameters.

30       Furthermore, because wafers have a finite thickness, the heat applied by the susceptor tends to generate another temperature gradient along the central axis of

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the wafer. Such axial gradients can both create and exacerbate the problems listed above.

Yet another temperature gradient typically exists between the rear surface of the substrate wafer and the front surface of the susceptor; *i.e.* a surface-to-surface gradient. It will thus be understood that both radiant and conductive heat transfer typically take place between susceptors and substrate wafers. Because many susceptors are formed of graphite coated with silicon carbide, the thermodynamic driving force created by the large temperature gradients between the susceptor and the silicon carbide wafers also causes the silicon carbide coating to undesirably sublime from the susceptor to the wafer.

Additionally, because such sublimation tends to promote pin hole formation in the susceptor coating, it can permit contaminants from the graphite to escape and unintentionally dope the substrates to the epilayers. This in turn ultimately leads to non-uniform doping levels in the semiconductor material, and reduces the lifetime of the susceptor. The problems created by susceptors which undesirably emit dopants is set forth for example in the background portion of U.S. Patent No. 5,119,540 to Kong et al.

Nevertheless, a need still exists for susceptors that can operate at the high temperatures required for silicon carbide processing while minimizing or eliminating these radial, axial and surface to surface temperature gradients, and the associated physical changes and problems.

#### **Object and Summary of the Invention**

Therefore, it is an object of the present invention to provide a susceptor for minimizing or eliminating radial, axial, and surface-to-surface thermal gradients across a substrate wafer.

The invention meets this object with a susceptor that comprises a first portion that includes a surface for receiving a semiconductor substrate wafer thereon, and a second portion facing the substrate receiving surface and spaced from the substrate receiving surface with the spacing being sufficiently large to permit the flow of gases therebetween for epitaxial growth on a substrate. The spacing remains small enough, however, for the second susceptor portion to heat

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the exposed face of a substrate to substantially the same temperature as the first susceptor portion heats the face of the substrate that is in direct contact with the substrate receiving surface.

In another aspect, the invention is a method for minimizing or eliminating thermal gradients in and around a substrate during epitaxial growth by heating a portion of a susceptor that faces, but avoids contact with, a semiconductor substrate, and that is spaced sufficiently far from the substrate to permit the flow of bases between the substrate and the susceptor portion to encourage epitaxial growth on the substrate facing the susceptor portion wherein the susceptor is thermally responsive to the irradiating radiation.

The foregoing and other object, advantages and features of the invention, and the manner in which the same are accomplished, will be more readily apparent upon consideration of the following detailed description of the invention taken in conjunction with the accompanying drawings, which illustrate preferred and exemplary embodiments, and wherein:

#### **Brief Description of the Drawings**

Figure 1 is a cross-sectional view of a platform type chemical vapor deposition (CVD) system:

Figure 2 is a cross-sectional view of a barrel-type CVD system;

Figure 3 is a graph illustrating the relationship between wafer deflection and wafer diameter at various temperature gradients;

Figure 4 is a schematic view of a barrel-type susceptor;

Figure 5 is a schematic view of wafer deflection and temperature gradients;

Figure 6 is a cross-sectional view of one embodiment of a susceptor according to the present invention;

Figure 7 is a partial cross-sectional view of a second embodiment of the susceptor of the present invention;

Figure 8 is a cross-sectional view of a pancake-type susceptor;

Figure 9 is a top plan view of a pancake-type susceptor according to the present invention.

### Detailed Description of the Preferred Embodiments

The present invention is a susceptor for minimizing or eliminating thermal gradients, including radial, axial, and surface-to-surface gradients, that affect a substrate wafer during epitaxial growth. Substrates according to the present invention are particularly useful for chemical vapor deposition systems as illustrated in figures 1 and 2. Figure 1 shows a platform or pancake type CVD system broadly designated at 20. The system comprises a reactor vessel 21 formed of a materials, typically a quartz tube or bell jar, that is substantially transparent to the appropriate frequencies of electromagnetic radiation. A gas supply system is in fluid communication with the reaction. A gas supply system is in fluid communication with the reaction vessel 21 and in Figure 1 is illustrated as the gas injector 22.

The system includes a source of electromagnetic radiation that in Figure 1 is illustrated as the induction coils 25. The operation of such generators and induction coils is generally well known to those of ordinary skill in the art, and will not be discussed further herein in detail. As is also understood in this art, alternative heating techniques can include electric resistance heating, radiant lamp heating, and similar techniques.

The chemical vapor deposition system shown in Figure 1 also includes the platform type susceptor 26 with semiconductor substrates, typically disc-shaped wafer 27 thereon. Figure 1 also illustrates the pumping port 30 for evacuating the system as desired.

Figure 2 illustrates a system that is very similar in terms of its basic operation, but that is a barrel-type susceptor, rather than a pancake-type. In Figure 2, the CVD system is broadly designated at 32 and shows a reaction vessel 33 which is surrounded by a water jacket 34 which circulates water against the walls of the reaction vessel 33. The CVD system 32 also includes a gas inlet 35 and a gas exhaust 36, a water inlet 37 and the water outlet 40, and a lifting and rotation assembly 41 for the susceptor.

The susceptor itself is broadly designated at 42 and is in the general shape of a cylinder although with a shallow slope that general shape of a cylinder,



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although with a shallow slope that gives it somewhat frustoconical shape. The cylinder is formed of a plurality of adjacent straight sidewall sections 43 that define the cylinder. A plurality of wafer pockets 44 are positioned on the sidewalls 43 and hold the semiconductor substrates thereon. The slight include of  
5 the susceptor walls help keep the wafers in the pockets 44, and improve the uniformity of the resulting epilayers by encouraging more favorable gas flow. Figure 2 also illustrates the power supply 45 for the induction coil broadly designated at 46.

Figure 3 is a graph that helps illustrate the problem addressed by the  
10 present invention. In Figure 3, the deflection of a wafer expressed in thousandths of an inch is plotted against the wafer diameter in inches for three different temperature gradients ("Delta T"). as noted in Figure 3, the susceptor surface temperature is 1530° C and the wafer thickness is 12 mils (0.012 inch). As Figure 3 illustrates, wafer deflection represents a minimal problem when the diameter of  
15 the substrate wafer is about an inch or less. For larger wafer, particularly those of two, three or even four inches, the deflection becomes more severe, even at relatively low temperature gradients.

Figure 4 illustrates a barrel type susceptor similar to that used in the illustration of Figure 2. Using the same numbering system as Figure 2, the  
20 susceptor is broadly designated at 42, is made of a plurality of straight sidewalls 43 that together define the generally cylindrical shape. The sidewalls 43 include a plurality of wafer pockets 44 for holding the substrate wafer.

Figure 5 is a schematic illustration of the effects of the temperature gradients plotted in Figure 3, and includes the designation of the axial temperature  
25 gradient ( $\Delta T_1$ ) and of the radial gradient ( $\Delta T_2$ ).

Figure 6 illustrates a susceptor according to the present invention that is most appropriately used in the barrel type systems illustrated in Figure 2. In the embodiment illustrated in Figure 6, the susceptor is broadly designated at 50 and is a cylinder formed of a plurality of adjacent straight sidewall sections 51. Figure 6  
30 illustrates two of the sidewalls in cross-section and one in side elevation. The straight sidewall sections 51, of which there are most typically four, six, or eight,

are formed of a material that is thermally responsive to selected frequencies of electromagnetic radiation. As noted above, the most common electromagnetic radiation is in the radio frequency range, so the susceptor material is generally selected to be thermally responsive to such RF frequencies. In preferred  
5   embodiments, the susceptor 50 is formed of graphite coated with silicon carbide.

        In a presently preferred embodiment, the electromagnetic radiation is applied in the 8-10 kilocycle range using a solid state power supply that takes advantage of the inherent efficiencies of solid state technology. Those familiar with inductive CVD processes will also recognize that thicker susceptor walls  
10   requires lower frequencies to achieve the most efficient penetration.

        In the embodiment illustrated in Figure 6, the susceptor 50 includes a plurality of wafer pockets 52 on the inner circumference of the cylinder. Thus, when the susceptor 50 is heated, the facing walls radiantly heat the front of the wafers while the susceptor heats the rear. As Figure 6 illustrates, in this  
15   embodiment, the sidewalls 51 preferably define an inverted truncated cone with a relatively shallow slope as compared to a true cylinder. As noted earlier, the shallow slope in the sidewalls 51 makes it somewhat easier to retain the wafers in the pockets 52 during chemical vapor deposition, and also helps provide a proper flow pattern for the CVD gases.

20         Figure 7 illustrates a next embodiment of the invention in which the susceptor comprises a first cylinder (or "barrel") broadly designated at 54. The cylinder is defined by a plurality of adjacent straight sidewall sections 55, and is formed of a material that is thermally responsive to selected frequencies of electromagnetic radiation. The cylinder 54 includes a plurality of wafer pockets 56  
25   on the outer surface of the sidewall sections 55.

        A second cylinder broadly designated at 57 surrounds the first cylinder 54 and defines an annular space A between the first and second cylinders. The second cylinder 57 is likewise made of a material that is thermally responsive to the selected frequencies of electromagnetic radiation, and the annular space between  
30   the first and second cylinders (54, 57) is sufficiently large to permit the flow of gases therebetween for epitaxial growth on substrates in the wafer pockets 56,

while small enough of the second cylinder 57 to heat the exposed face of substrates to substantially the same temperature as the first cylinder 54 heats the faces of substrates that are direct contact with the first cylinder.

The first and second cylinders 54, 57 can be formed of either the same or  
5 different materials. If used in a barrel type susceptor system as illustrated in Figure 2, the second cylinder 57 tends to heat the first cylinder 54 to encourage the cylinders to reach substantially the same temperatures. As in other embodiments, each of the cylinders is most preferably formed of graphite coated with silicon carbide.

10 It will be understood that the use of a silicon carbide coating on such susceptors is a function of the ceramic properties of polycrystalline silicon carbide and is otherwise not related to its semiconductor properties. Thus, susceptors made of stainless steel, graphite, graphite coated with silicon carbide, or silicon carbide, are typically used in the semiconductor industry for CVD processes.

15 Figures 8, 9 and 10 illustrate another susceptor according to the present invention. Figures 8 and 9 illustrate in cross-section and top plan view respectively, a pancake or plate-shaped susceptor broadly designated at 60. The susceptor 60 has a top surface 61 for receiving semiconductor substrate wafers thereon. In this embodiment, the invention further comprises a horizontally  
20 disposed second susceptor portion 63 parallel to and above the wafer receiving surface 61 of the first susceptor portion 60. Both of the susceptor portions 60 and 63 are formed of materials that are thermally responsive to selected frequencies of electromagnetic radiation, and as in the previous embodiments, are preferably formed of the same material to be responsive the same frequencies of  
25 electromagnetic radiation, and as in the previous embodiments, are preferably formed of the same material to be responsive to the same frequencies of electromagnetic radiation. Most preferably, both susceptor portions 60 and 63 are formed of graphite coated with silicon carbide. As in the previous embodiments, the spacing designated B (Figure 10) between the two portion 60, 63 is sufficiently  
30 large to permit the flow of gases therebetween for epitaxial growth on a substrate on the surface 61, while small enough for the second susceptor portion 63 to heat

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the exposed face of a substrate to substantially the same temperature as the first susceptor portion 60 heats the face of substrate that is in direct contact with the substrate receiving surface 61. As illustrated in Figures 8, 9, and 10, the top surface 61 of the first horizontal susceptor portion 60 preferably includes a plurality of wafer pockets 64.

In each of these embodiments, it will be understood that the two susceptor portions can be connected to one another, or separate portions of a single susceptor, or independent pieces as may be desired or necessary under various circumstances. Additionally, the optimum spacing between the substrate portions can be determined by computer modeling or actual practice, and without requiring undue experimentation.

In another aspect, the invention comprises a method for minimizing or eliminating thermal gradients in a substrate during epitaxial growth. In this aspect, the invention comprises irradiating a susceptor, or a susceptor portion, that faces, but avoids contact with, a semiconductor substrate wafer, and that is spaced sufficiently far from the wafer to permit the flow of gases between the substrate and the facing susceptor to thereby encourage epitaxial growth on the substrate facing the susceptor portion. As in the structural embodiments, the susceptor is thermally responsive to the irradiating radiation.

As further set forth with respect to the structural aspects of the invention, the invention also preferably comprises concurrently irradiating a separate susceptor portion upon which the wafer rests so that the exposed face of the substrate is heated to substantially the same temperature as is the face of the substrate that is in direct contact with the other susceptor portion.

The method further comprises the steps of directing source gases that flow between the heated susceptor portions. If the epitaxial are to be formed of silicon carbide, the method preferably comprises directing silicon and carbon containing source gases such as silane, ethylene, and propane.

Where other materials, such as Group III nitrides, are to form the epitaxial layers on the silicon carbide, the step of directing sources gases can include

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directing gases such as trimethyl aluminum, trimethyl gallium, trimethyl indium, and ammonia.

In preferred embodiments, the method also comprises the step of preparing the substrate surface for growth. As set forth in more detail in the references  
5 incorporate above, such preparation can comprise steps such as oxidizing the surface followed by a chemical etching step to remove the oxidized portion leaving a prepared surface behind, or alternatively, dry etching the silicon carbide surface to prepare it for further growth. As in most epitaxial growth technique, surface preparation further typically comprises lapping and polishing the substrate surface  
10 prior to the oxidation or etching steps.

In the drawings and specifications, there have been disclosed typically preferred embodiments of the invention and, although specific terms have been employed, they have been used in a generic sense and in descriptive sense only, and not for purposes of limitation, the scope of the invention being set forth in the  
15 following claims:

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**CLAIMS:**

1. A susceptor for minimizing or eliminating thermal gradients that affect a substrate wafer during epitaxial growth, said susceptor comprising:
  - a first susceptor portion including a surface for receiving a semiconductor
  - 5 substrate wafer thereon; and
  - a second susceptor portion;
  - characterized in that:
    - said second susceptor portion faces said substrate receiving surface and is
    - spaced from said substrate-receiving surface, said spacing being sufficiently large
    - 10 to permit the flow of gases therebetween for epitaxial growth on a substrate on said
    - surface, while small enough for said second susceptor portion to heat the exposed
    - face of a substrate to substantially the same temperature as said first susceptor
    - portion heats the face of a substrate that is in direct contact with said
    - substrate-receiving surface.
- 15 2. A chemical vapor deposition system comprising:
  - a reactor vessel formed of a material substantially transparent to
  - electromagnetic radiation;
  - a gas supply system in fluid communication with said reactor vessel;
  - 20 a source of electromagnetic radiation external to said reaction vessel; and
  - a susceptor according to Claim 1 within said reaction vessel.
3. A chemical vapor deposition system according to Claim 2 wherein said
- 25 reaction vessel is made of quartz.
4. A chemical vapor deposition system according to Claim 2 wherein said
- reaction vessel is made of stainless steel.
5. A chemical vapor deposition system according to Claim 2 wherein said
- 30 source of electromagnetic radiation comprises an induction coil surrounding said
- reaction vessel.

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6. A susceptor according to Claim 1 or Claim 2 wherein said first susceptor portion is formed of a material that is thermally responsive to electromagnetic radiation.

5           7. A susceptor according to Claim 1 or Claim 2 wherein said second susceptor portion is formed of a material that is thermally responsive to electromagnetic radiation.

8. A susceptor according to Claim 1 or Claim 2 wherein said first and  
10 second portions are formed of the same material.

9. A susceptor according to Claim 1 or Claim 2 wherein said first and second portions are formed of different materials.

15           10. A susceptor according to Claim 1 or Claim 2 wherein said first and second susceptor portions are formed of graphite coated with silicon carbide.

11. A susceptor according to Claim 1 or Claim 2 wherein said substrate receiving surface further comprises a plurality of wafer pockets.  
20

12. A susceptor according to Claim 1 or Claim 2 wherein said first and second portions together define a cylinder formed of a plurality of adjacent straight sidewall sections that define the cylinder, with a plurality of wafer pockets on the inner circumference of said cylinder.

25

13. A susceptor according to Claim 12 wherein said sidewalls define an inverted truncated cone.

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14. A susceptor according to Claim 1 or Claim 2 wherein:

said first portion comprises a first cylinder formed of a plurality of adjacent straight sidewall sections that define the cylinder, said cylinder being formed of a material that is thermally responsive to selected frequencies of electromagnetic radiation and having a plurality of wafer pockets on the outer surface of said  
5 sidewall sections; and

said second portion comprises a second cylinder surrounding said first cylinder and defining an annular space between said first and second cylinders, said second cylinder being made of a material that is thermally responsive to  
10 selected frequencies of electromagnetic radiation, with the annular space between said first and second cylinders being sufficiently large to permit the flow of gases therebetween for epitaxial growth on substrates in said wafer pockets, while small enough for said second cylinder to heat the exposed face of substrates to substantially the same temperature as said first cylinder heats the faces of  
15 substrates that are in direct contact with said first cylinder.

15. A susceptor according to Claim 1 or Claim 2 wherein said first susceptor portion is horizontally oriented and formed of a material that is thermally responsive to selected frequencies of electromagnetic radiation, and has a top  
20 surface for receiving semiconductor substrate wafers thereon; and

a second susceptor portion is parallel to and spaced apart from said wafer-receiving surface of said first susceptor portion and formed of a material that is thermally responsive to selected frequencies of electromagnetic radiation, said spacing being sufficiently large to permit the flow of gases therebetween for  
25 epitaxial growth on a substrate on said surface, while small enough for said second susceptor portion to heat the exposed face of a substrate to substantially the same temperature as said first susceptor portion heats the face of a substrate that is in direct contact with said substrate-receiving surface.

30 16. A susceptor according to Claim 15 wherein said top surface of said first susceptor portion includes a plurality of wafer pockets.



17. A method for minimizing or eliminating thermal gradients that affect a substrate during epitaxial growth, the method comprising:

heating a portion of a susceptor that faces, but avoids contact with, a semiconductor substrate and that is spaced sufficiently far from the substrate to  
5 permit the flow of gases between the substrate and the susceptor portion to encourage epitaxial growth on the substrate facing the susceptor portion.

18. A method according to Claim 17 and further comprising concurrently heating a second susceptor portion upon which the wafer rests so that the exposed  
10 face of the substrate is heated to substantially the same temperature as is the face of the substrate that is in direct contact with the second susceptor portion.

19. A method according to Claim 17 wherein the heating step comprises irradiating a susceptor that is thermally responsive to certain frequencies of the  
15 electromagnetic radiation with electromagnetic radiation within the range of those certain frequencies.

20. A method according to Claim 17 and further comprising the step of directing source gases to flow between the heated susceptor portions.

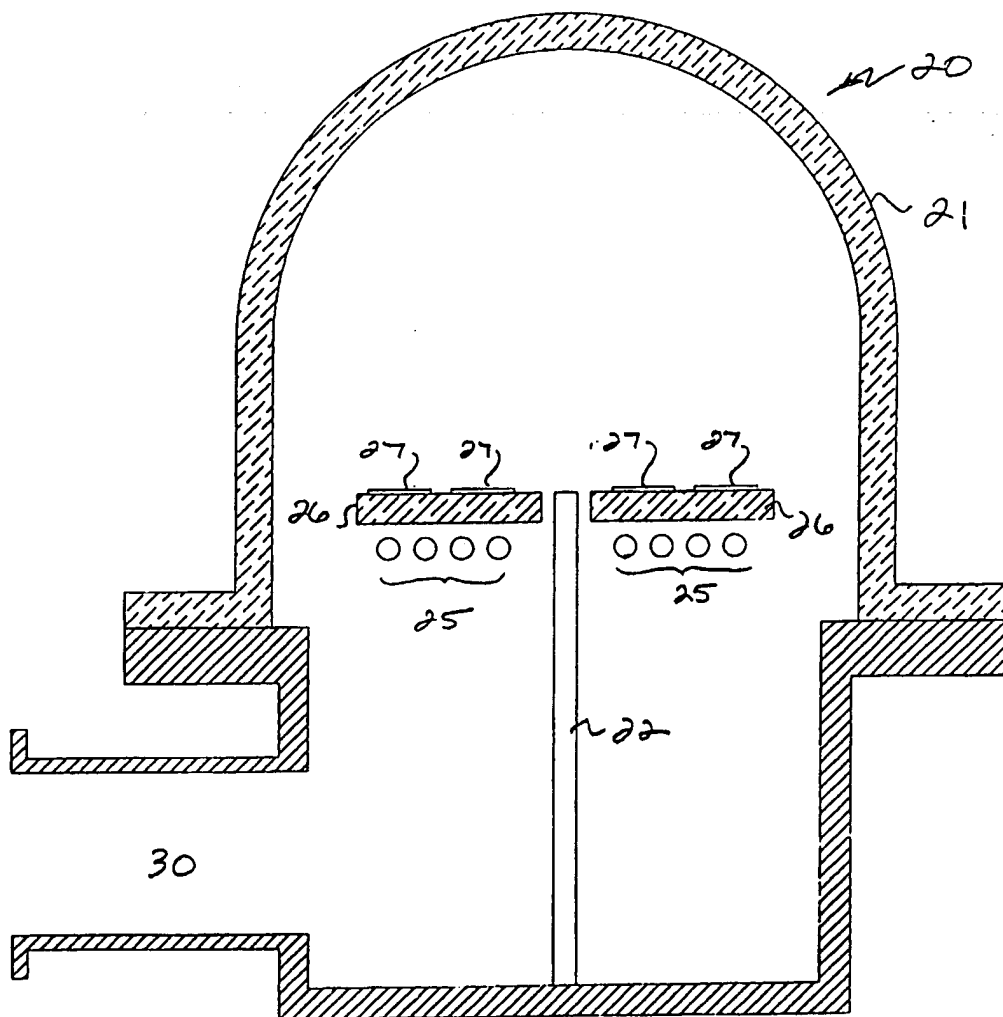


Figure 1

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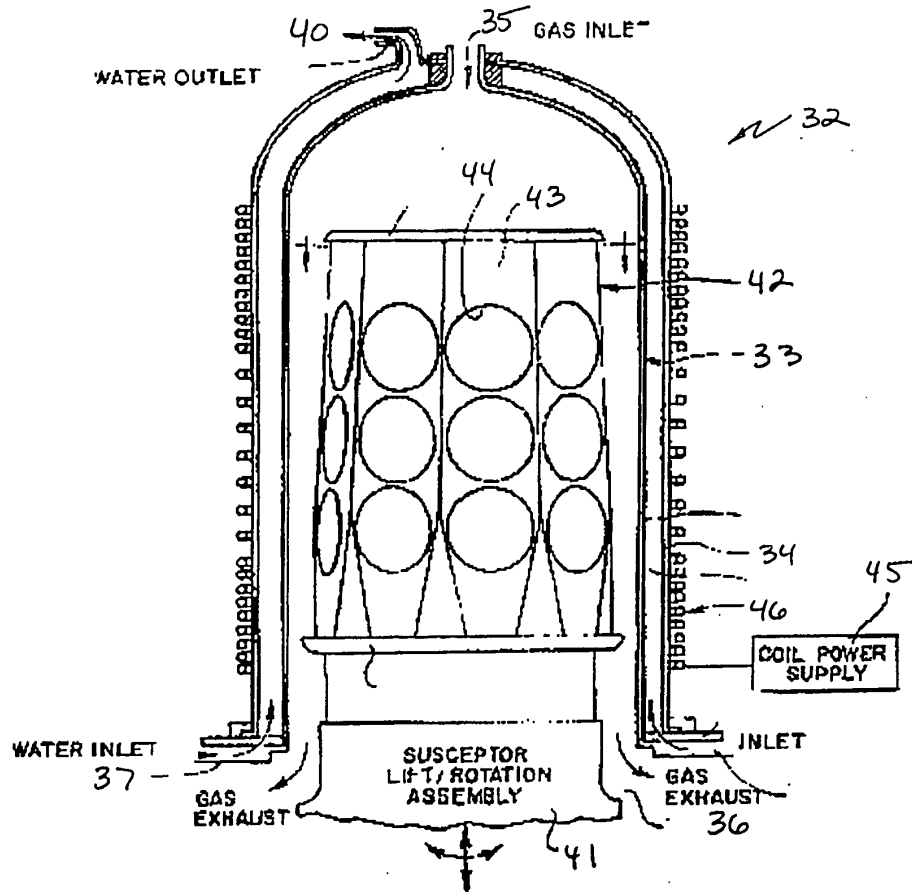


Figure 2

Figure 3

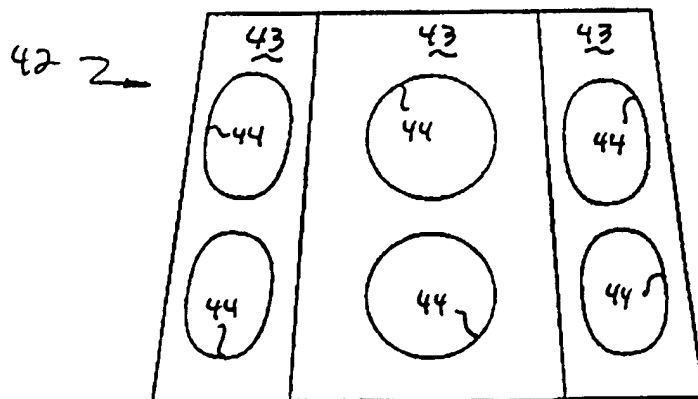
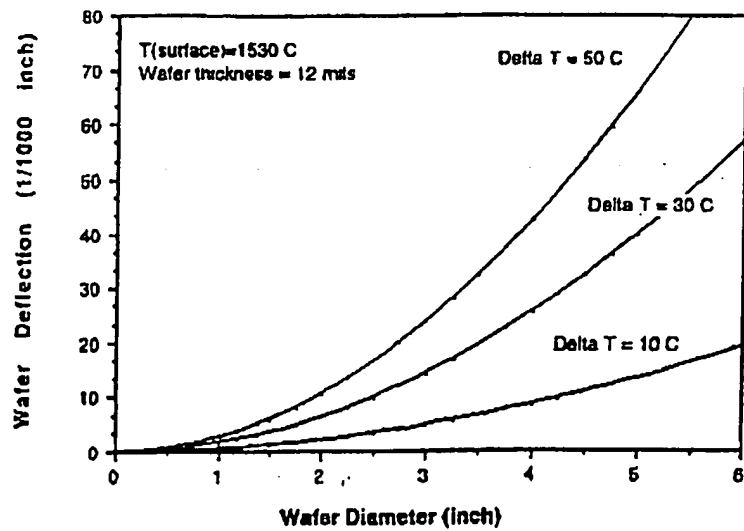


Figure 4

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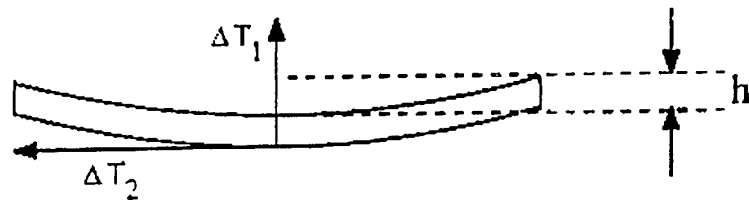


Figure 5

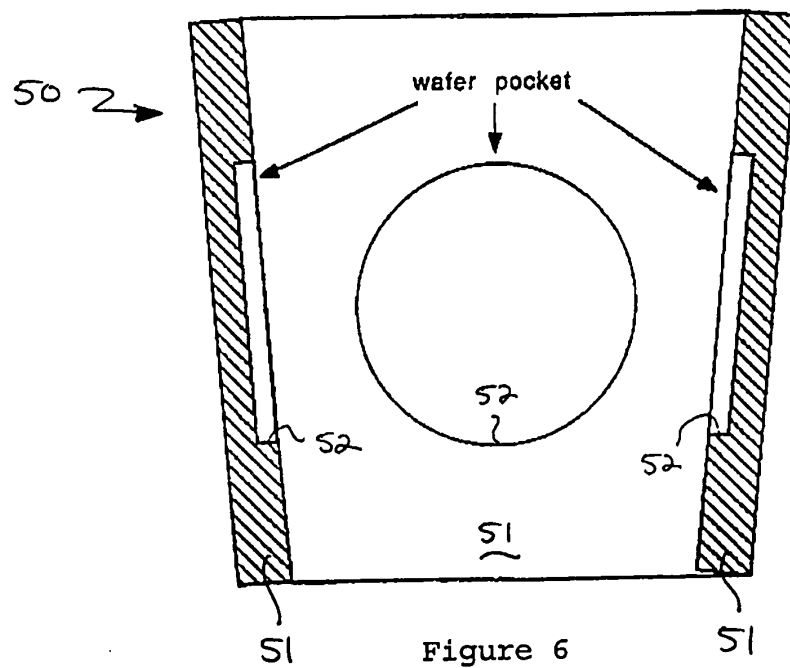


Figure 6

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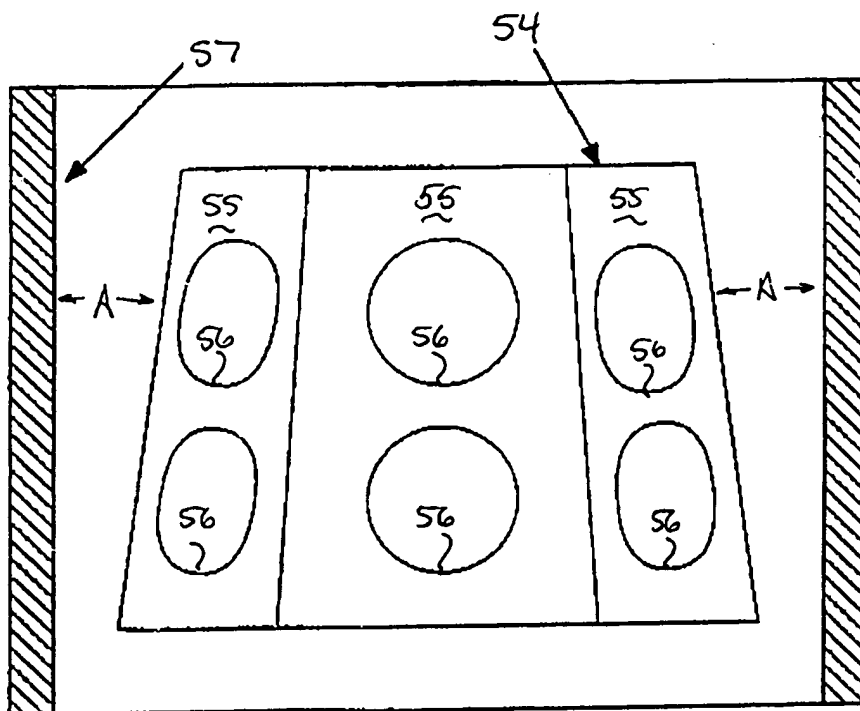


Figure 7

Figure 8

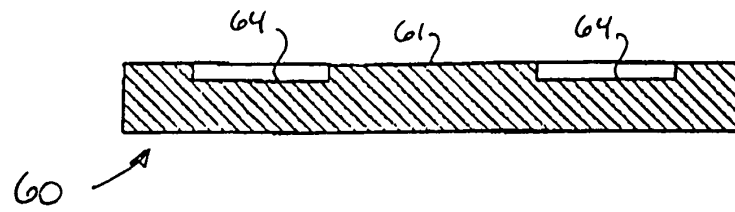


Figure 9

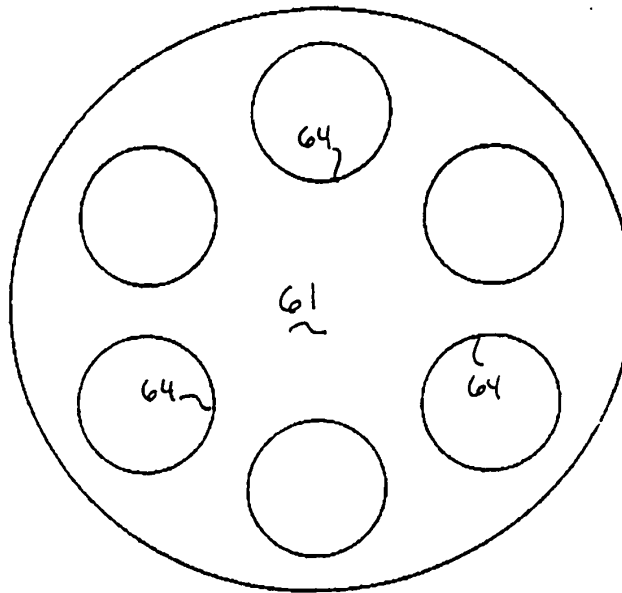
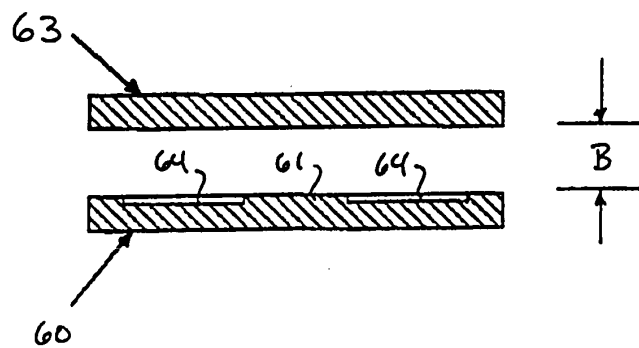


Figure 10



# INTERNATIONAL SEARCH REPORT

Int lonal Application No

PCT/US 98/05484

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C30B25/12 C23C16/44

According to International Patent Classification(IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C30B C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 330 708 A (NIPPON KOKAN KK) 6 September 1989  see column 2, line 35 - column 5, line 28; figure 1 -----	1-3,5-7, 12-14, 17-20
X	EP 0 258 052 A (MASSACHUSETTS INST TECHNOLOGY) 2 March 1988  see column 3, line 45 - column 6, line 15; figure 19 see column 12, line 15 - column 14, line 12 -----	1-3,5-8, 11,12, 14,17-20
X	US 4 186 684 A (GORMAN RALPH ET AL) 5 February 1980 see column 3, line 32 - column 6, line 45; figures 2,6 -----	1,17,18, 20

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

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"&" document member of the same patent family

Date of the actual completion of the international search

5 June 1998

Date of mailing of the international search report

16/06/1998

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 98/05484

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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US 4186684	A	05-02-1980	NONE	

